Progress Report on PVC Formulations and Extrusion

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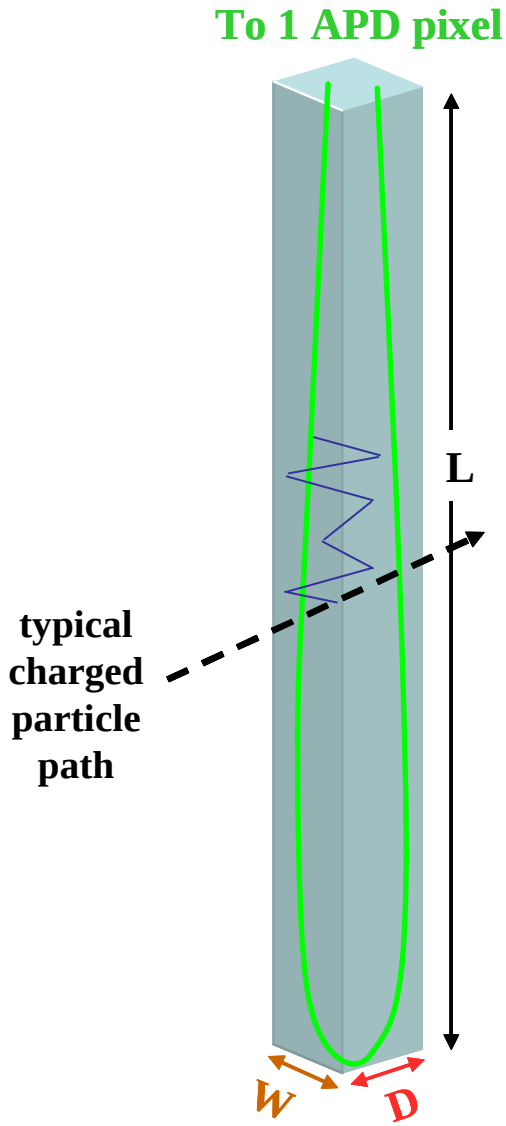
NOνA Working Group Meeting
October 4, 2006

The Basic Detector Element



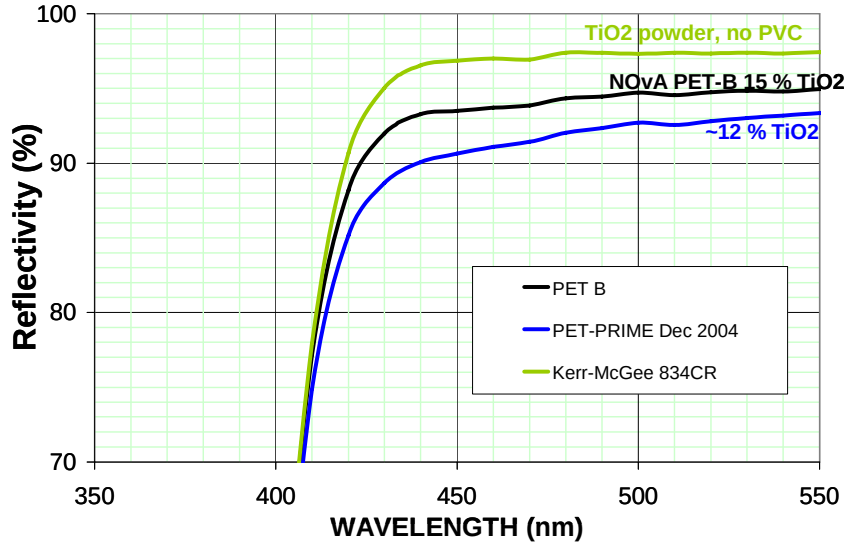
- Liquid Scintillator
  - 5.5% pseudocumene as scintillant
  - Mineral oil and waveshifters (PPO, bis-MSB)
- **PVC cell for primary containment**
  - Highly reflective titanium dioxide walls
  - Diffuse reflection keeps light local to track along the cell length to ± 25 cm
- Looped wavelength shifting fiber to collect light
  - 0.8 mm diameter, double clad, K27 waveshifter
- Avalanche Photodiode
  - 85% quantum efficiency @ 500 – 550 nm
- Low noise amplifier

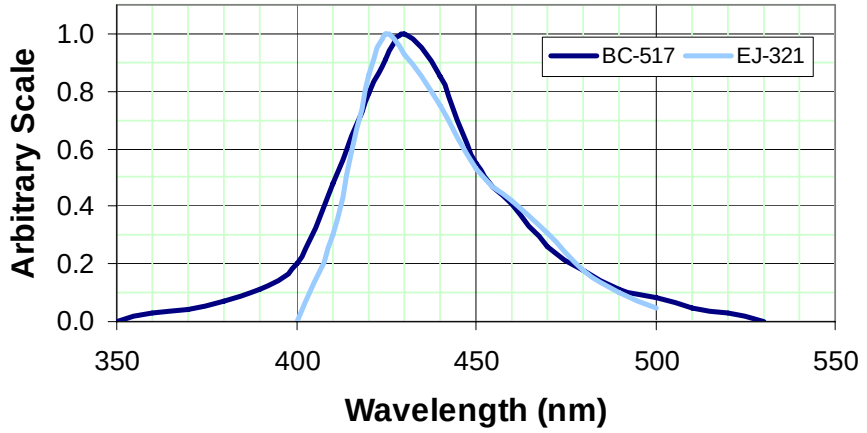
Scintillation Spectrum & Reflectivity

Reflectivity:

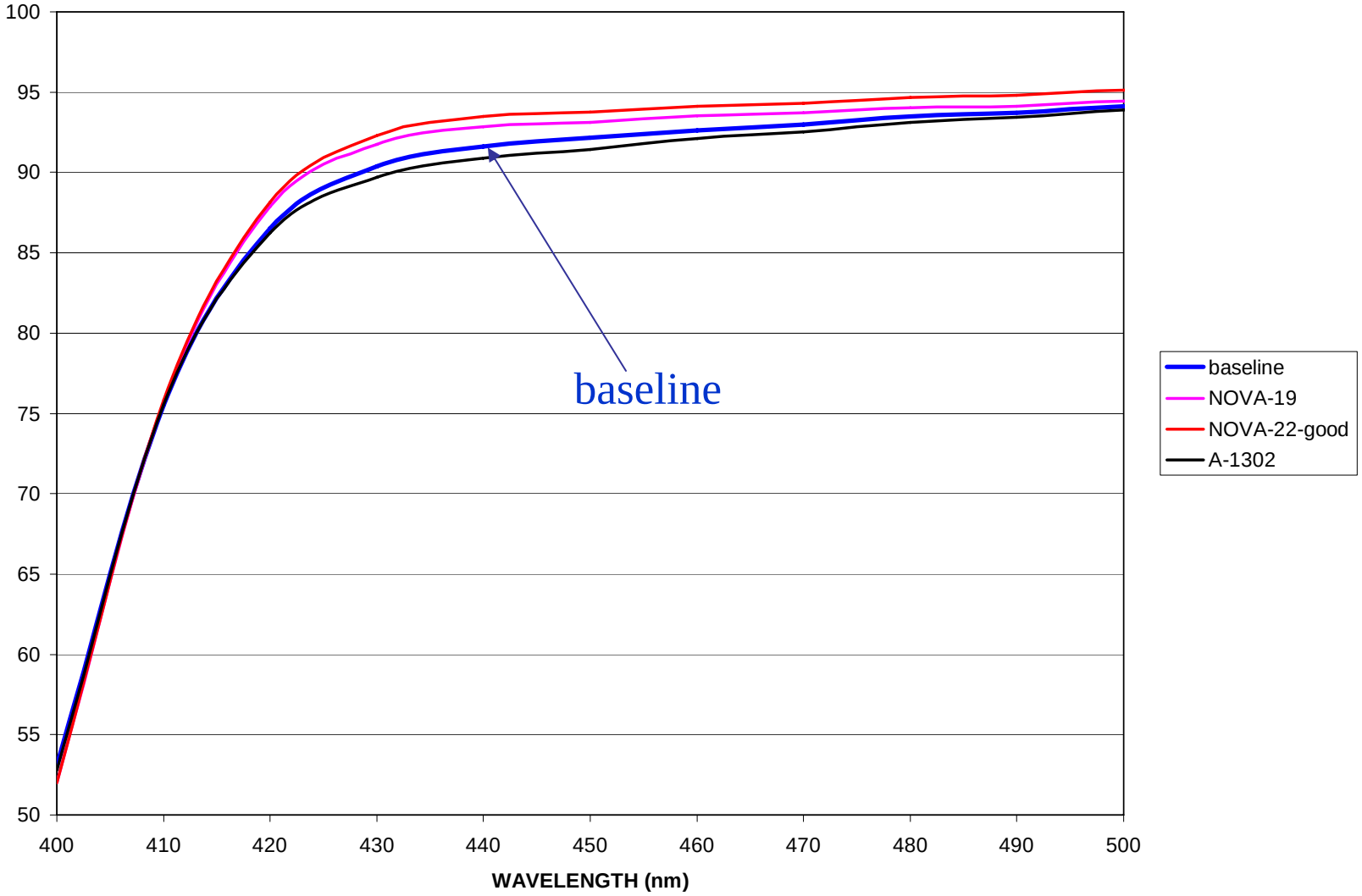
Rutile TiO_2

Scintillation Spectrum





Typical Reflectivity Curves

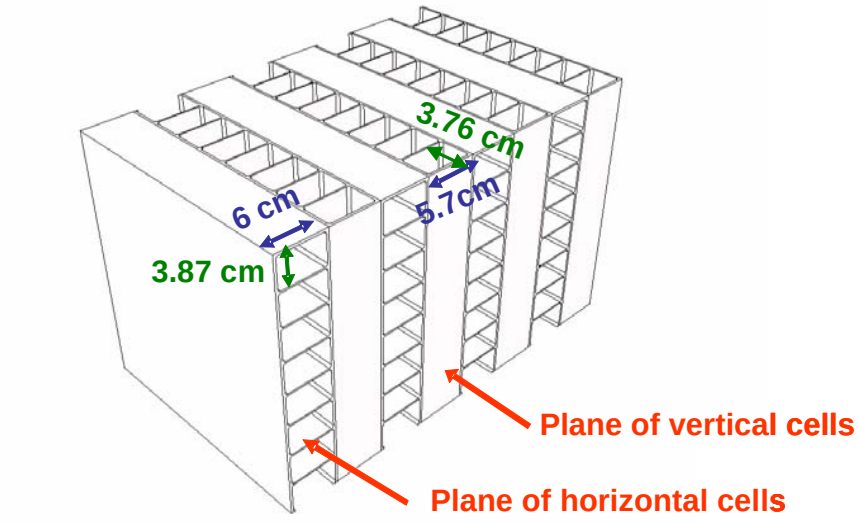


PVC Cellular Extrusions

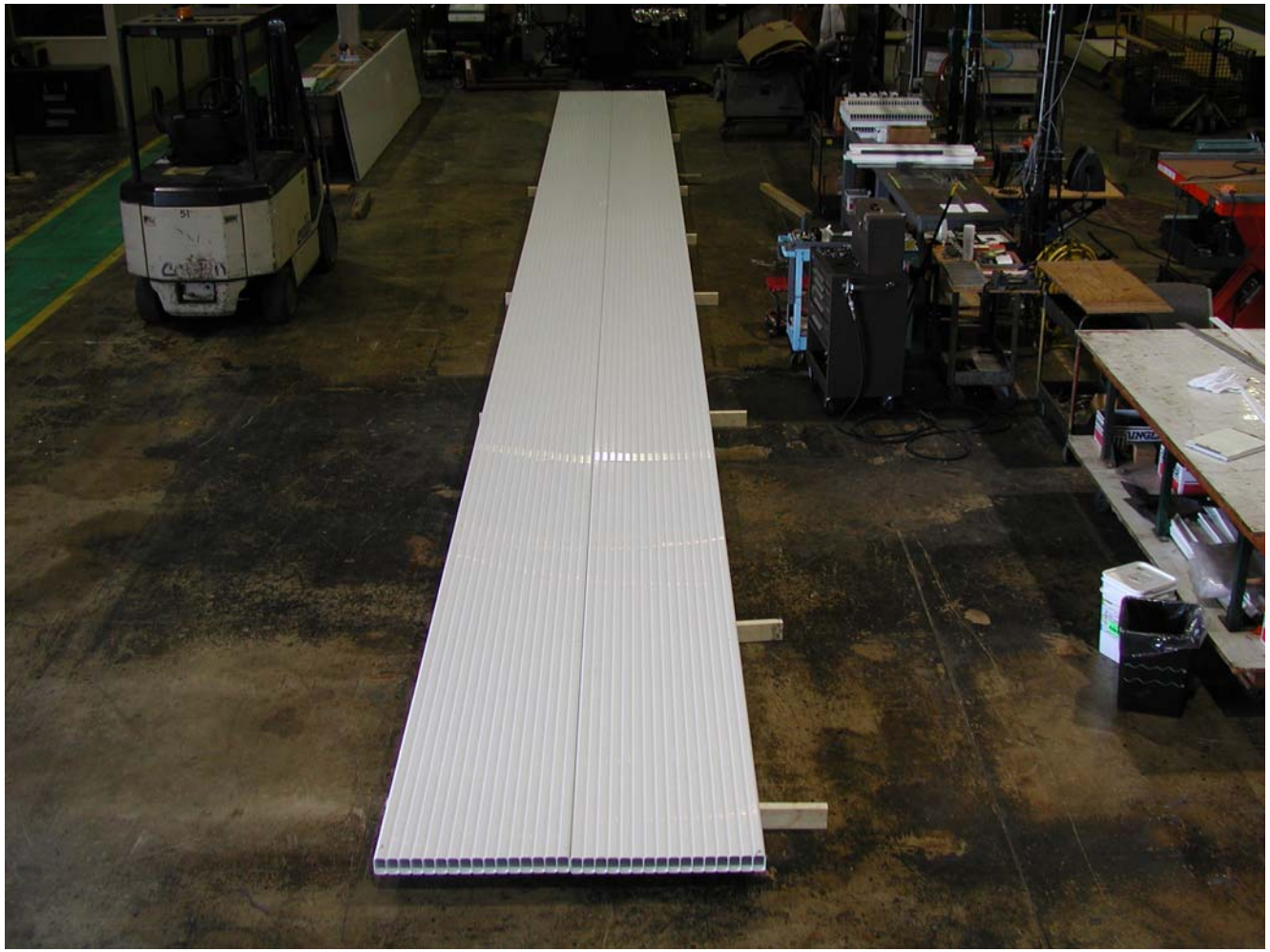
15.5 meter extrusion made on 1st test of NOvA 16-cell die June 31, 2006

Assemble into a Detector

- Modules are made from two extrusions
  - Two 16-cells glued side-to-side
  - 32 cells ➔ 32 channels
    - 32 is the basic electronic readout unit
- Detector Plane is 12 modules wide
  - 15.5 m x 15.5m plane
- Bond the planes together into **Blocks**
  31 planes thick
  - Alternate Vertical & Horizontal Planes
  - All horizontal modules are supported by vertical planes
    - Vertical modules have thicker walls
- Far Detector consist of 44 **Blocks**



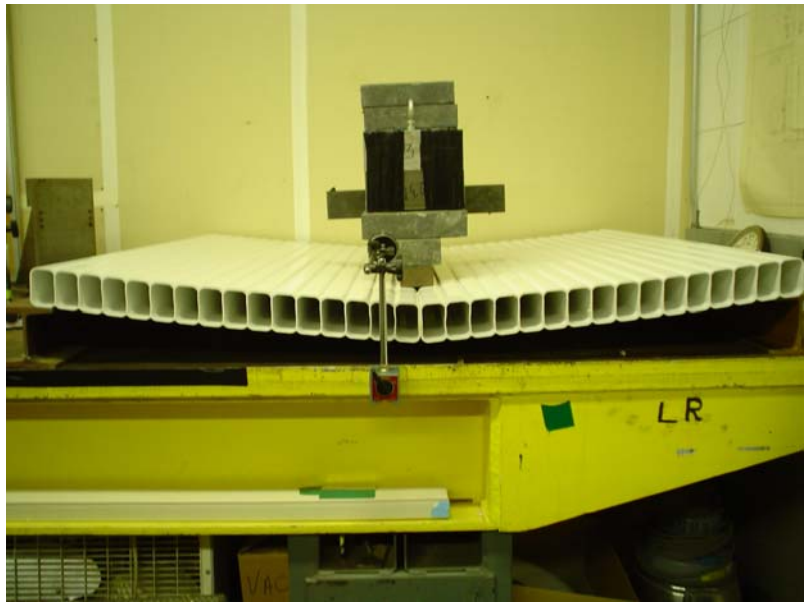
Two 15.5 m extrusions per module



From 1st test production; not glued together

Two 16-Cell Extrusions Glued

- **NOvA Detectors will have modules with 32 cells**
- 32-cell extrusions may be difficult to make
  - –Expensive development work for very wide die and tooling is needed
- 32-cell modules can be made by gluing two 16-cell extrusions



- Glue joint finally failed with load of 100 pounds per foot

"Devcon Plastic Welder"

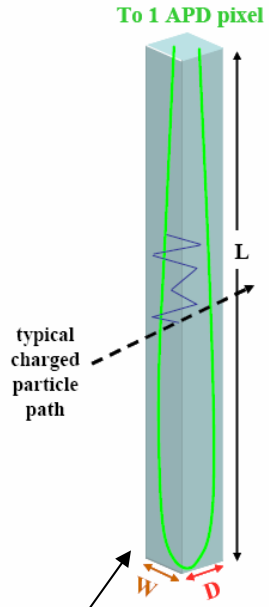
Horizontal and Vertical Extrusions

*Vertical Extrusions have **thicker walls***

External Dimensions are Identical

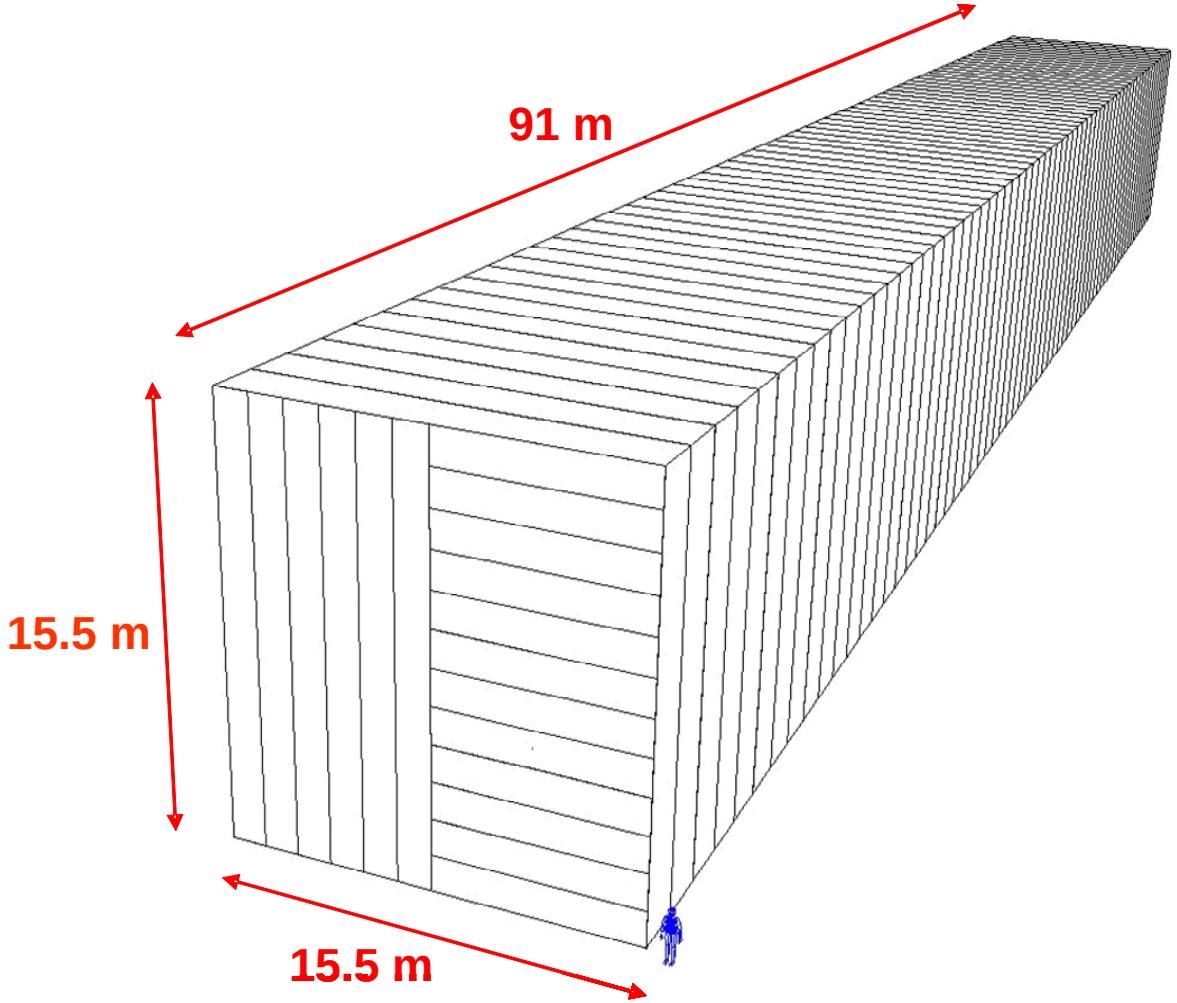
This Makes Assembled Blocks Stronger

- Horizontal Extrusions
  - **38.7 mm x 60 mm cells** (open area)
  - **3 mm** outer wall
  - **2 mm** web thickness
  - ~ **300 lbs** for 15.5m
- Vertical Extrusions
  - **37.6 mm x 57 mm cells** (open area)
  - **4.5 mm** outer wall
  - **3 mm** web thickness
  - ~ **450 lbs** for 15.5 m



Hydrostatic pressure 19.2 psi

Full Detector composed of 44 blocks



- 44 blocks (each 127 metric tons of PVC)
  - 1364 planes form a structure 91 meters long

PVC WBS 1.4 Team

- Level 3 managers
  - Chuck Grozis, FNAL (extruding)
  - Jim Grudzinski, ANL (PVC resin, extruded PVC mechanical properties)
  - Karen Kephart, FNAL (shipping and handling)
- Project Chemist
  - Anna Pla-Dalmau FNAL (extruding, PVC resin)

PVC: Two Major Functions

- Detector Building Block: mechanically strong
  - Vertical and Horizontal Extrusions
    - Verticals have 50% thicker walls than horizontals
  - Modules are Made by Joining Two Extrusions
    - Then “add fibers and seal the ends”
  - Detectors: Bond Modules to Make Rigid Units
    - 31-plane “Blocks” (far detector)
    - 7 and 8 –plane “Segments” (near detector)
- Light Collector: reflective inner surfaces
  - Scintillation photons bounce an average of 10 times before entering WLS fiber
    - $(\text{reflectivity})^{10}$ ➔ small changes in reflectivity have large impact
  - Requires high reflectivity in range ~ 400-470 nm

R & D Goals

1. Develop and/or identify a PVC resin suitable for NOvA
   - Reflectivity
   - Strength
   - Entire formula is known by NOvA PVC team (desirable)
2. Develop 16-cell Prototype Die and Downstream Sizing to NOvA Specifications (*horizontal and vertical* extrusion dimensions)
   - Matched to a suitable extruder and production line
   - Matched to a NOvA-selected PVC resin
3. Determine Properties of Extruded PVC
   - Reflectivity (light yield)
   - Mechanical Strength & Durability
4. Produce high-quality PVC for NOvA Prototypes
   - Integration Prototype Near Detector (IPND) and Full-Scale Far Detector Prototypes

1. PVC Resin

- **PVC resin** must extrude very well
  - Continuous smooth flow (slip) and no sticking to the die
  - No yellowing (heat buildup; several possible mechanisms)
- **Extruded PVC** must have consistent properties
  - High reflectivity of scintillation light
  - Good mechanical properties after extruding: good strength and minimal creep properties
  - Cellular structure must meet tolerances: +/- 0.25 mm wall thickness and +/- 0.5 mm web thickness over entire 15.7 m length

PVC Resin Composition

- **PVC resins**
  - ~80% - 90% PVC + ~ 10 *other ingredients*
- Concern that *other ingredients* may absorb light
  - ***But several of those ingredients are necessary for the extrusion process***
- **"NOvA PET-B": Baseline PVC Resin**
  - Based on early R & D with **P**lastics **E**xtrusion **T**echnologies
  - The number of ingredients was limited to maximize reflectivity
    - (Attempt to **exclude** ingredients that absorb blue light)

NOvA PET-B Custom Formulation: what we specified

- 14.8% TiO2 rutile crystal (for reflectivity)
- 82.5% PVC resin (typically about 50% of the cost)
- 0.8% Lubricants (provide release from metals and nearby PVC in extruding)
- 1.9% Organo-Tin Stabilizers (prevent breakdown when PVC is subjected to heat and shear)

- ***But the complete NOvA PET-B formulation is unknown to us***
  - PVC resin manufacturers will not reveal their formulas
- NOvA hired a consultant to develop PVC resin with all ingredients known to us

PVC Resin, continued

- Ongoing R&D program to improve PVC resin
  - Strength and reflectivity
  - TiO_2 is responsible for reflectivity
    - **Rutile crystal-** commonly used with PVC
    - **Anatase crystal-** not common in PVC products
      - Offers higher reflectivity below 420 nm
      - Must be used in larger proportions
    - ***Increased reflectivity can reduce other costs: Scintillator & Fiber***
- Two PVC compounding companies have produced their own resin mixtures with rutile and anatase crystalline forms of TiO_2
  - **Both companies have responded to an RFP to make NOvA resin**
    - Aurora Plastics
    - Prime PVC (their proprietary formulations have the better reflectivity)
- NOvA consultant James Summers designs custom PVC resins
  - Compounding companies do not disclose formulae if *they* develop PVC resin
  - Compounding companies ***will make a resin*** to NOvA specifications if we specify all ingredients ➔ much larger pool of possible vendors ➔ lower cost

Initial NOvA Resins Designed by James Summers
Extrusions were made with a Tabletop Extruder

NOvA-2, for example

		phr	1	2	3	4	5	6	7	8	9	10	11	12
PVC	Shintech SE950W or Shintech SE950EG or equivalent in color & thermal stability		100	100	100	100	100	100	100	100	100	100	100	100
Tin stabilizer	Rohm & Haas Advastab TM-182 80% monomethyl tin or equivalent		2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Titanium dioxide rutile	DuPont R-102 or equivalent in brightness & blue tone		12	19	0	18	19	19	19	18	0	19	19	0
Titanium dioxide rutile	Tronox CR-834 bright and blue tone - good		0	0	0	0	0	0	0	0	18	0	0	18
Titanium dioxide anatase	Millennium Tiona AT-1		0	0	19	0	0	0	0	0	0	0	0	0
Calcium stearate	Crompton Calcium stearate F now Chemtura Corp. or equivalent		0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Paraffin wax	Crompton Sunolite 160 now Chemtura Corp. or equivalent		1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	0.6
Oxidized polyethylene	Honeywell AC 629A or equivalent		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.1
Glycerol monostearate	Lonza Glycolube 825 or equivalent		0.5	0.5	0.5	0.5	0.5	0	1.0	0.5	0.5	0.5	0.5	0
Acrylic impact modifier	Rohm & Haas Paraloid KM 334 or equivalent		5	5	5	0	0	5	5	0	0	5	5	0
MBS impact modifier	Rohm & Haas Paraloid BTA 751-L or equivalent		0	0	0	0	4	0	0	0	0	0	0	0
Processing aid	Rohm & Haas Paraloid K120N or equivalent		1.0	1.0	1.0	1.0	1.0	1.0	1.0	0.0	0.0	1.0	1.0	0.0
		Total phr	123	130	130	124	129	129	130	123	123	130	130	122
		wt % titanium dioxide	10	15	15	15	15	15	15	15	15	15	15	15
Twin screw	Brabender Extrusion													
Achieve T by screw rpm	Melt Temperature, ^{o}C.		199	199	199	199	199	199	199	199	199	207	182	199
	Melt Temperature, ^{o}F.		390	390	390	390	390	390	390	390	390	405	360	390

PVC Resins Extruded in Recent Tests

- PET B Formulations made by Prime
  - 15% rutile, 18% rutile — no significant difference in reflectivity
  - 15% anatase, 18% anatase
    - **Reflectivity extended to shorted wavelengths**; 18% better than 15%
- Aurora Formulations
  - 15% rutile proprietary compounds have ~ 2-3% less reflectivity than baseline (rutile)
- NOvA Formulations

Extrutech's 6-cell die June '06

- NOvA-2 (prime) * selected for RFP
- NOvA-2 (aurora)
- NOvA-4 (aurora)
- NOvA-7 (aurora)
- NOvA-13 (aurora) anatase
- NOvA-16 (prime)

NOvA 16 cell die July and September

NOvA-2 (prime) burn streaks in this die
NOvA-19 (prime) * best rutile
NOvA-21 (aurora) anatase
NOvA-22 (prime) rutile
NOvA-23 (aurora) * best anatase
- same formula as NOvA-22 except TiO2

- Size of die has impact on "extrudability"; burn streaks due to longer residence time in die
- NOvA-2 needed more lubrication ➔ NOvA-19 and NOvA-22; NOvA-21 and NOvA-23
- NOvA-19 and NOvA-23 need slight modification: reduce lubricants

PVC Resin: Lessons Learned

- NOvA-2 was under-lubricated for 16-cell die
  - PVC stuck to die, heated and burned
  - Caused burn streaks on extrusion (occasional thin brown lines)
  - Eventually would plug the die
- NOvA-21, -22, -23, -19 were over-lubricated
  - PVC did not stick to itself very well
    - Poor "knitting" of cell dividers
    - To overcome this problem, PVC was extruded at the upper limit of temperature range ~385-390 F
  - PVC did not burn…even when extruder ran very slowly due to operator error
- Reduce lubricants slightly to operate nearer to middle of extruding machine's operating range
  - NOvA-19 ➔ NOvA-24 (rutile)
  - NOvA-23 ➔ NOvA-25 (anatase)

Most Recent NOvA Resins

	phr	Nova 2 for reference	Nova 19 for reference	Nova 21 for reference	Nova 22 for reference	Nova 23 for reference	**New Nova 24**	**New Nova 25**
PVC	Shintech SE950EG (high reflectivity)	100	100	100	100	100	**100**	**100**
Tin stabilizer	Rohm & Haas Advastab TM-182 80% monomethyl tin	2.5	0	0	2.5	2.5	**0**	**2.5**
Tin stabilizer	Rohm & Haas Advastab TM-181 20% monomethyl tin	0	2.5	2.5	0	0	**2.5**	**0**
Titanium dioxide rutile	DuPont R-102 rutile titanium dioxide (bright & blue tint)	19	19		19		**19**	
Anatase titanium dioxide				24		24		**24**
Calcium stearate	Ferro 15F calcium stearate	0.3	0.8	0.8	0.8	0.8	**0.8**	**0.7**
Paraffin wax	Ferro 165 paraffin wax	1.0	1.2	1.2	1.2	1.2	**1.1**	**1.1**
Oxidized polyethylene	Ferro Petrac 215 oxidized polyethylene	0.2	0.2	0.2	0.2	0.2	**0.2**	**0.2**
Glycerol monostearate	Rohm & Haas F1005 glycerol monostearate	0.5	0.4	0.4	0.4	0.4	**0.3**	**0.3**
Acrylic impact modifier	Rohm & Haas Paraloid KM 334 Acrylic impact modifier	5	0	0	4	4	**0**	**4**
Acrylic impact modifier	Arkema Durastrength 200 Acrylic impact modifier	0	4	4	0	0	**4**	**0**
Processing aid	Rohm & Haas Paraloid K120N processing aid	1.0	1.0	1.0	1.0	1.0	**1.0**	**1.0**
	wt % titanium dioxide	15	15	18	15	18	**15**	**18**

Next (and last?) PVC Resins

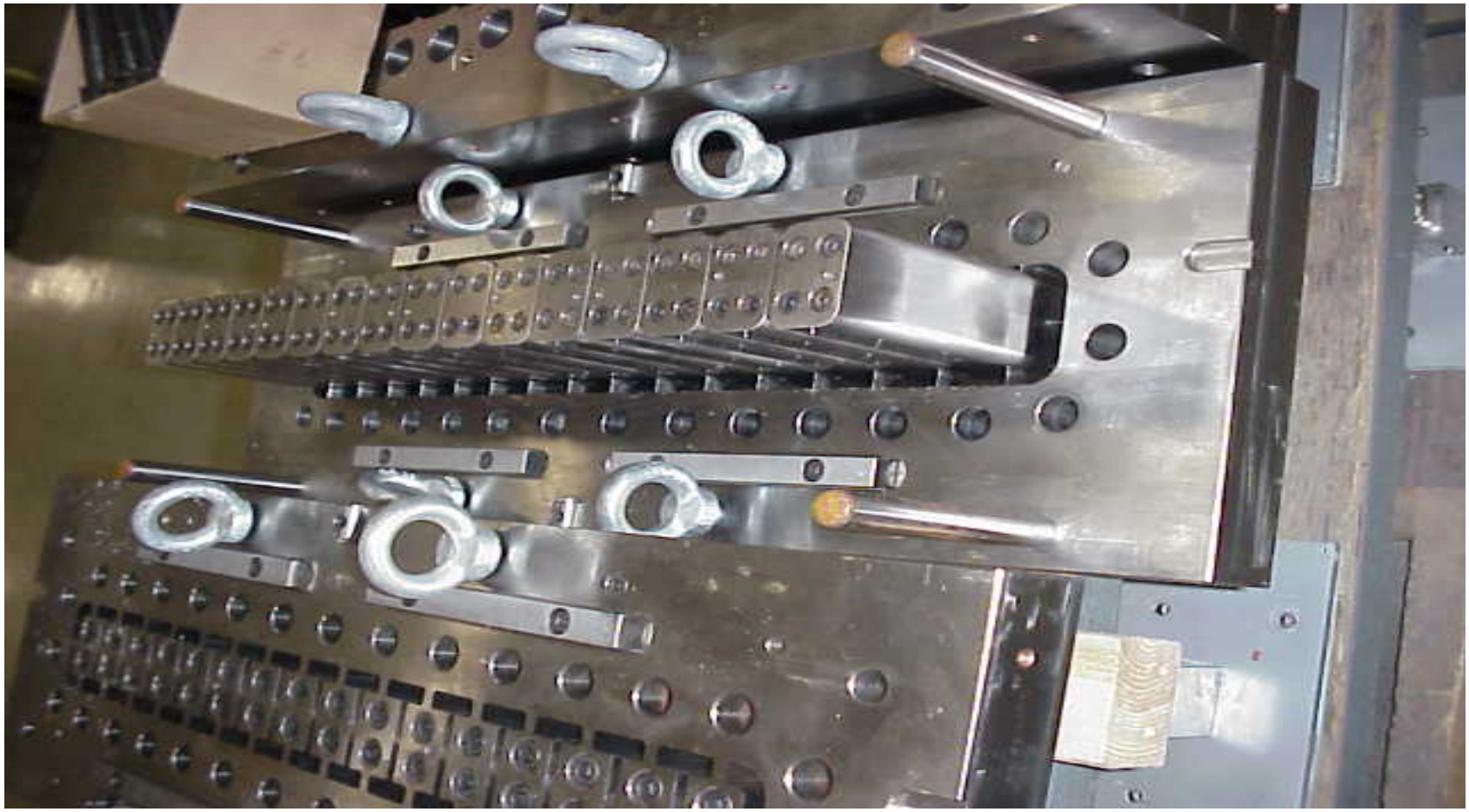
- NOvA-24 15% rutile
- NOvA-25 18% anatase

- Order 12,000 lbs NOvA-24
  - Tune the die and machine parameters to meet NOvA specifications
  - Produce good working samples for NOvA
- Order 6,000 lbs NOvA-25
  - Tune machine parameters only to produce the best anatase samples for NOvA
- Target extruding date: week of October 23 ’06

2. NOvA 16-Cell Prototype Die

- December, 2005 Placed Order for 16-cell die and tooling
  - Extrutech Plastics, Inc.
    - Selected by RFP Process
    - **Contract includes tuning the die/tooling to produce extrusions that meet NOvA tolerance limits**
    - **Tuning will be done once NOvA selects the PVC resin it wants to use**
- First tests of die and tooling
  - July/August 2006
    - Standard commercial PVC resin
      - extrusions close to tolerance limits
    - NOvA-2 PVC resin (R&D) made good extrusions but also stuck to the die
  - September 2006
    - NOvA-19, NOvA-22 rutile resins
    - NOvA-21, NOvA-23 anatase resins
    - Aurora 1290 commercial resin & 1302 Resin made for NOvA

16-cell Die: Interior

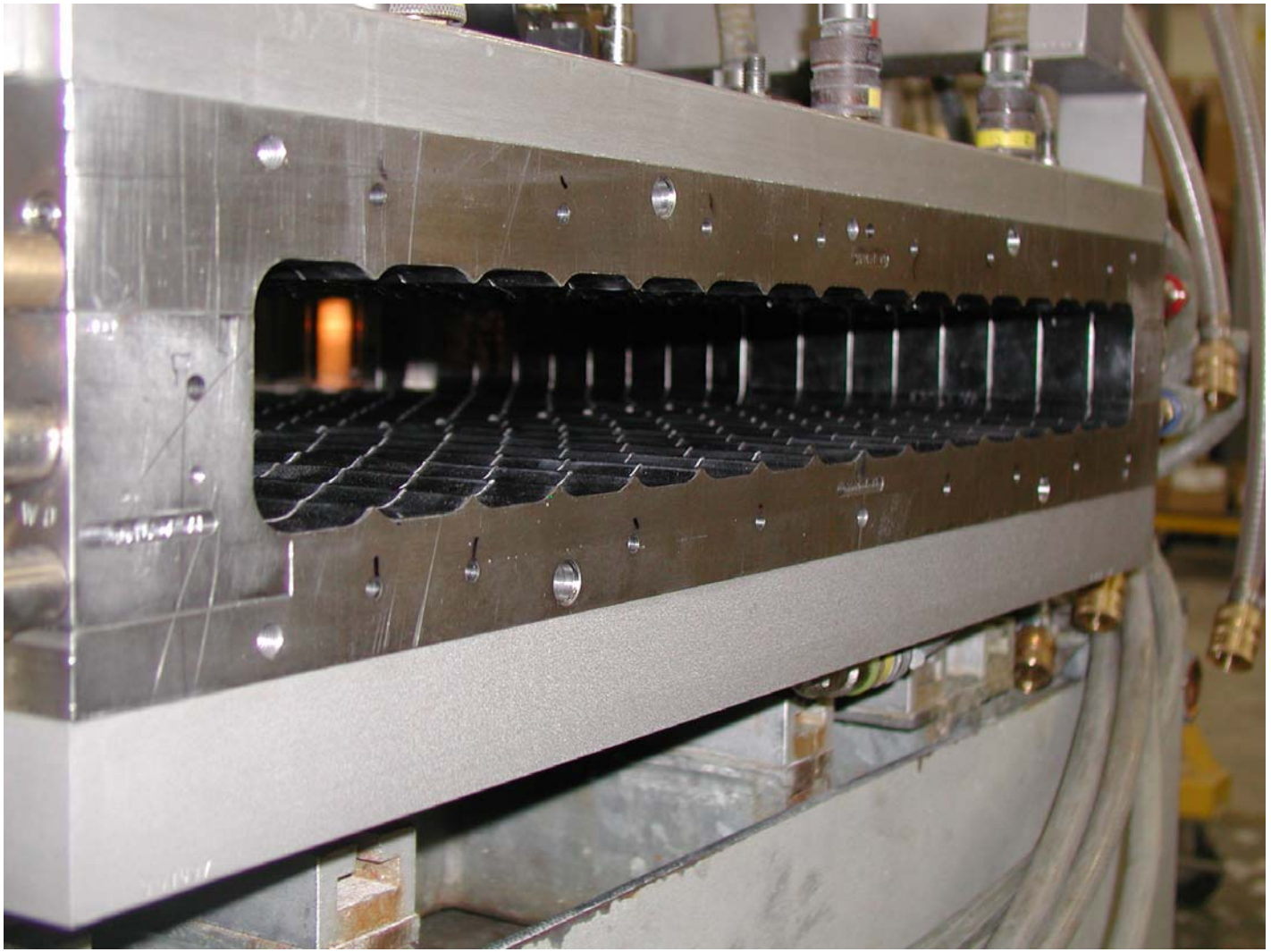


Die Attached to Extruder

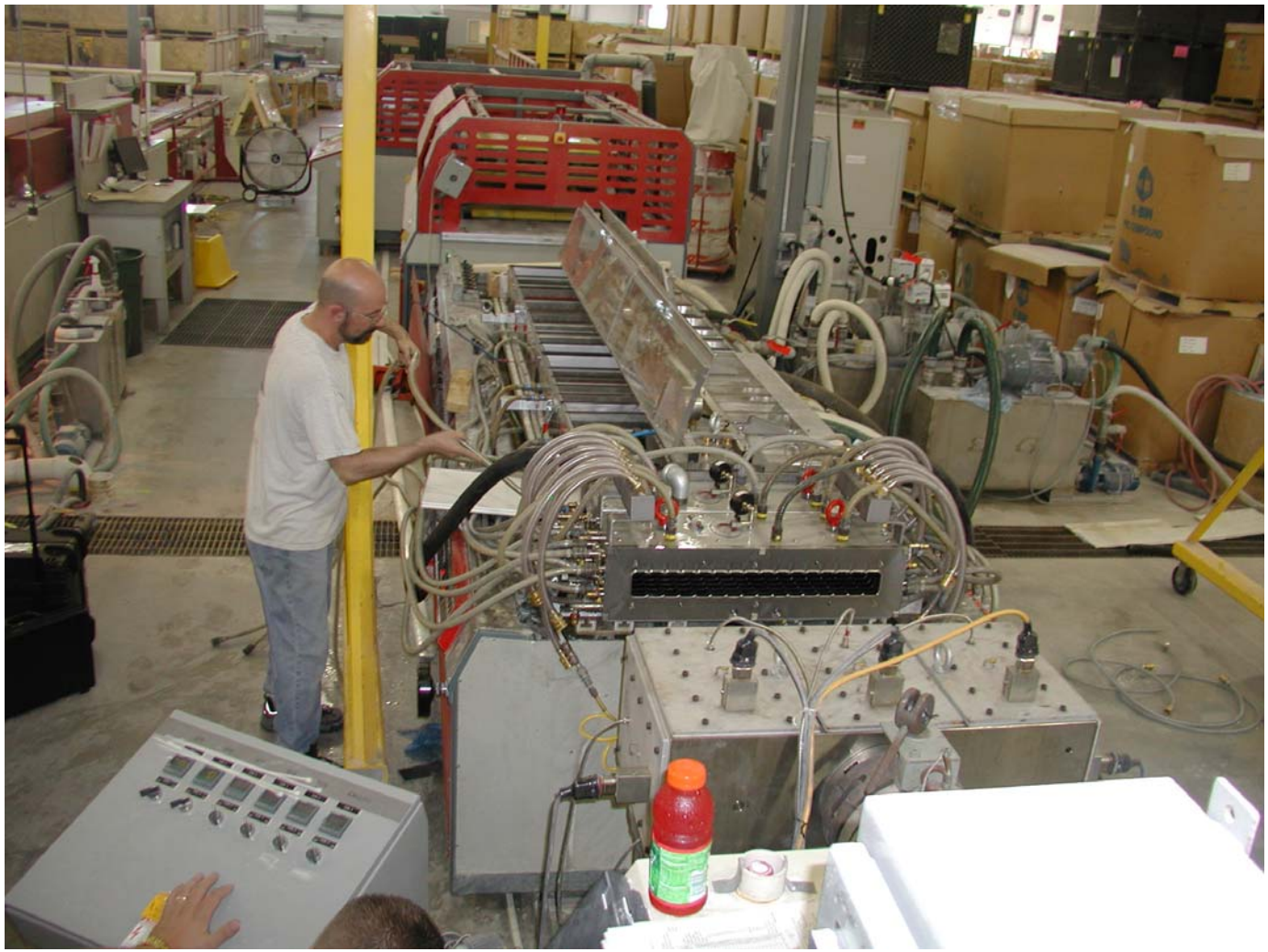
16 cell prototype die: plan

- Die will be tuned for Horizontal Extrusion production
  - October-November ’06
- Produce Horizontal Extrusions in December ’06, including total…
  - Integration Prototype Near Detector
  - Far Detector Full Scale Prototypes
- Extrutech/Greiner are producing new die inserts to modify 16-cell die for Vertical Extrusion production
  - Fabrication Oct.-Dec. ‘06
  - Remove Horizontal Inserts and replace with Vertical Inserts January ‘07
- Die will be tuned for Vertical Extrusion production
  - February – March 2007
- Produce Vertical Extrusions in April 2007, including total…
  - Integration Prototype Near Detector
  - Far Detector Full Scale Prototypes
- Re-install Horizontal Inserts and see if die still works well without further tuning (summer 2007)
  - Potential cost saving in production phase

Downstream Sizing



Die, Sizing, Cooling Tank and Puller

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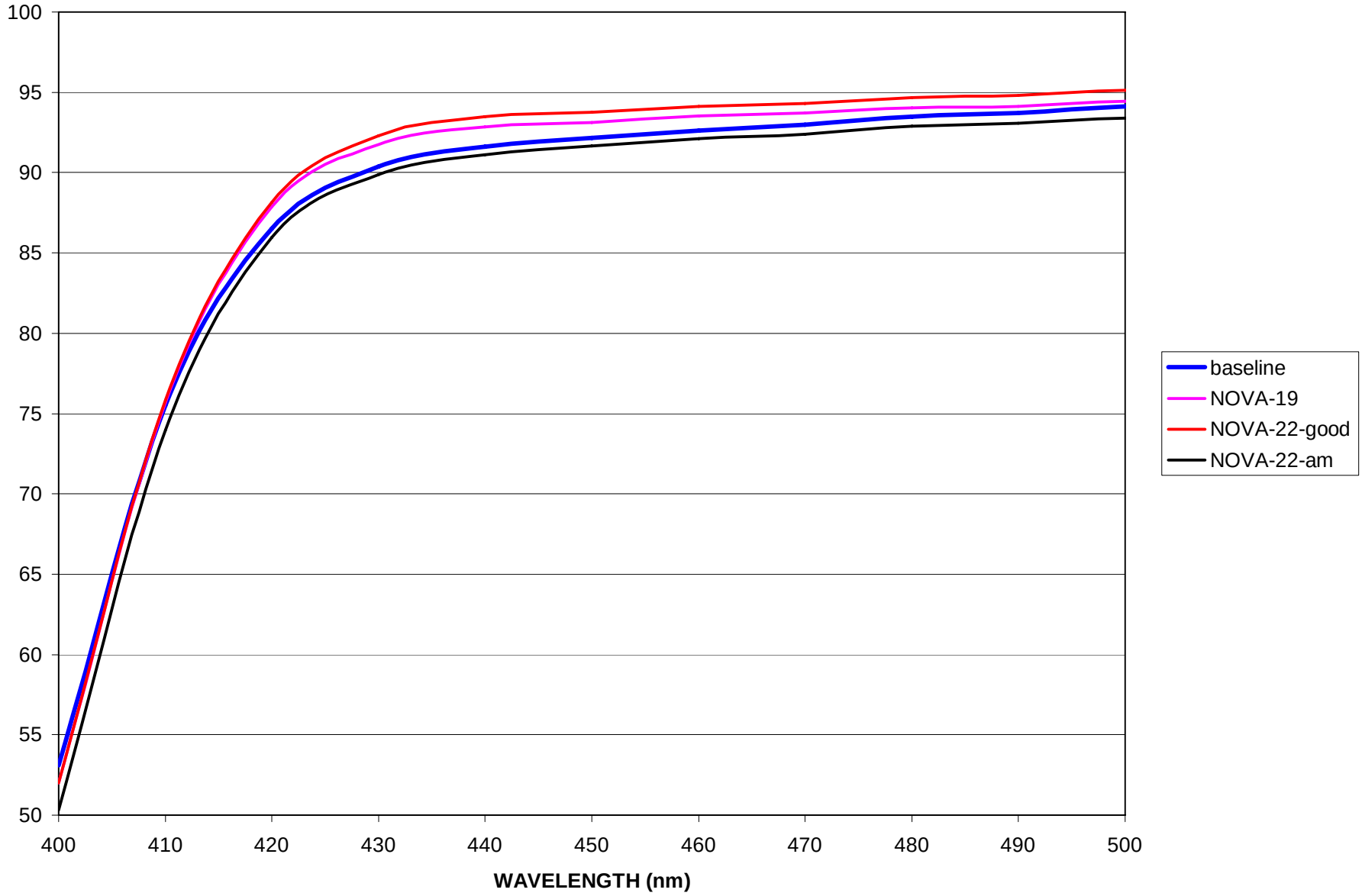
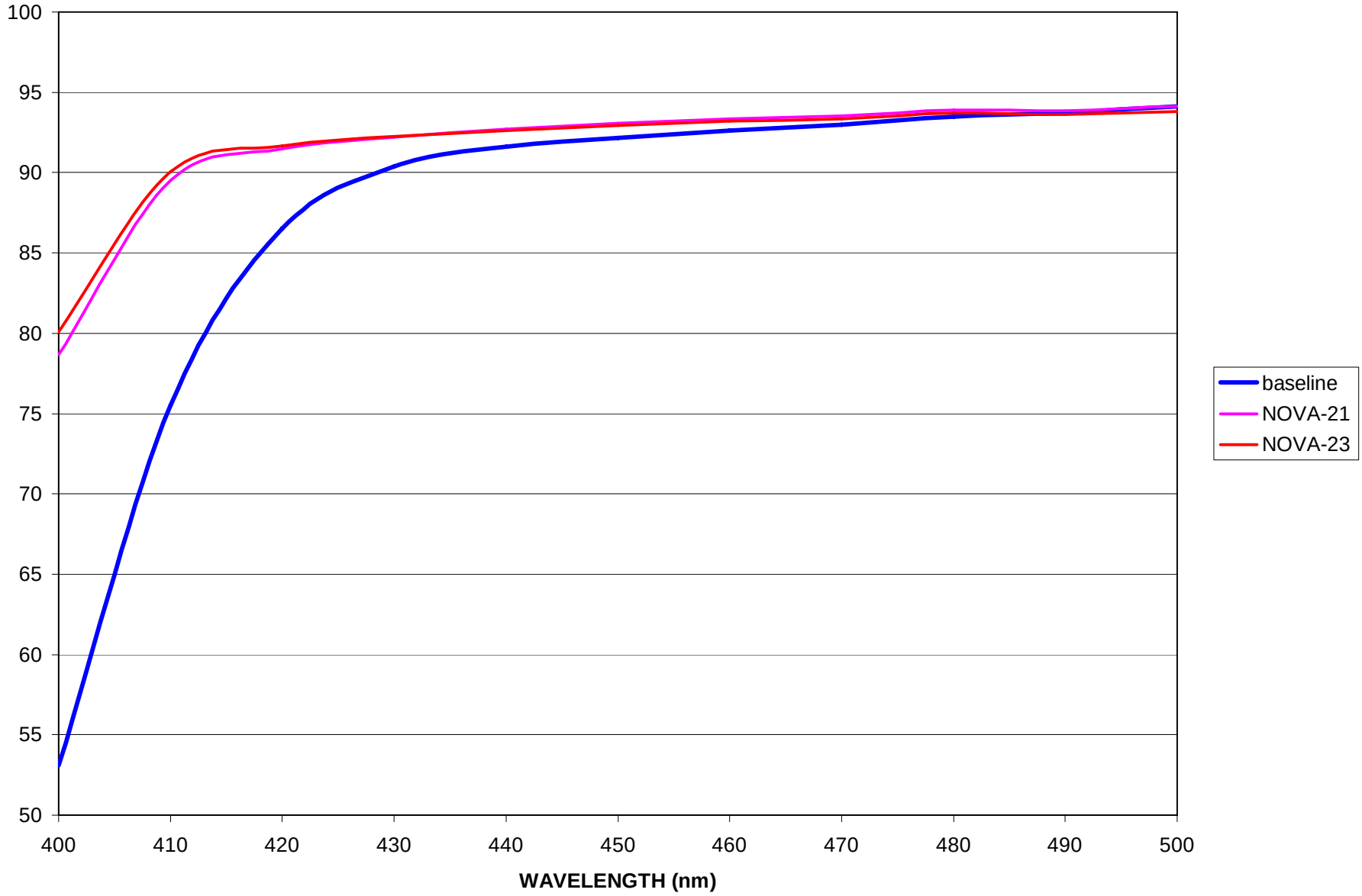
3. Physical Properties: Reflectivity

- Reflectivity is related to light yield
  - Rutile-to-rutile comparisons
  - Anatase-to-anatase comparisons
- Light yield is measured with
  - Liquid scintillator
  - WLS fiber
  - Extruded cell
- Anatase PVC yields ~10-15% more light than rutile
  - More definitive tests are in progress
  - Next round of extruding will add to this information

NOVA 19 & 22: 15% RUTILE



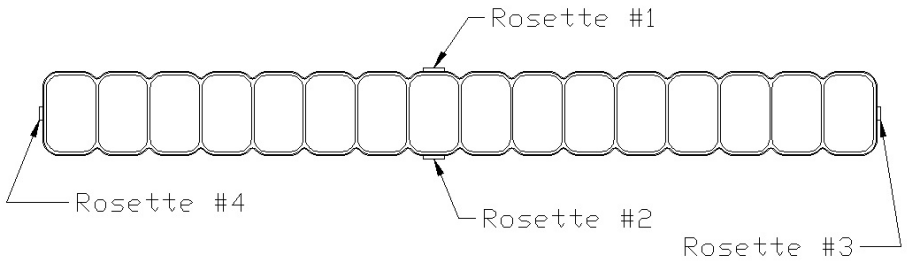
NOVA 21 & 23: 18% ANATASE



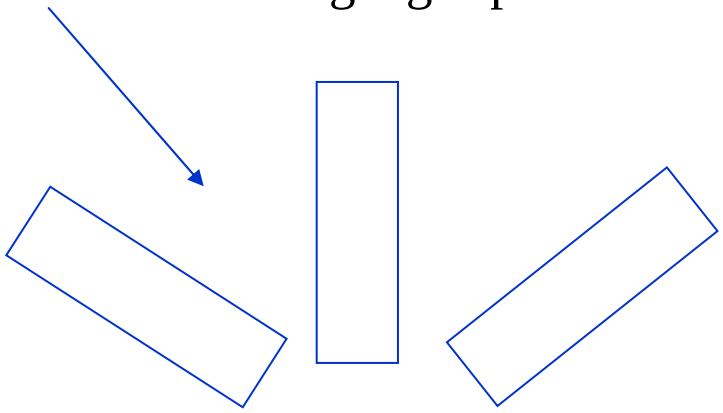
Structural Testing: Measurements

Pressurize 4-ft long Extrusion to 19 psi

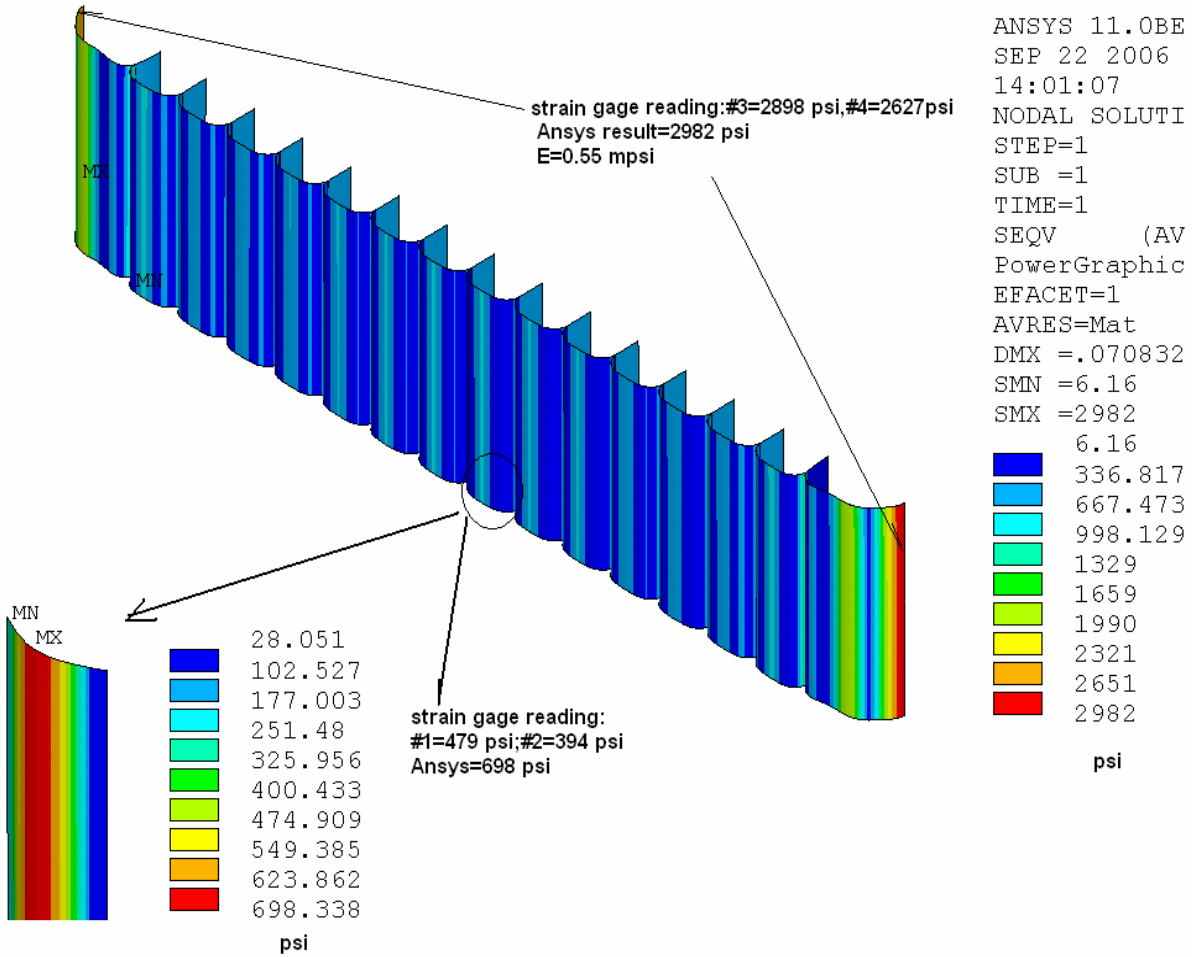
Simulates the hydrostatic pressure at bottom of vertical extrusion



Rosette = three strain gauges placed 45 degrees apart



Structural Testing: Finite Element Analysis



Comparison of Measurements and FEA

	Test Results		FEA Model	
	Stress psi	Strain (Micro)	Stress psi	Strain (Micro)
Scallop side	**480**	872	**694**	1270
60 mm side	**2900**	5270	**2982**	5422

- Excellent Agreement on the 60 mm flat side: 2982 psi vs. 2900 psi
- More difficult measurement of strain on scalloped side due to size of gauges compared to changes in the material's shape; agreement to ~ 30%

4. Prototype Extrusions for NOvA

- Six 15.5 m extrusions have been made (2,000 lbs)
  - 5 to U. Minnesota
  - 1 to Indiana U.
- IPND-size extrusions have been delivered (5,000 lbs)
  - ANL and Minnesota
- More extrusions are expected this month
  - Improved machine tune & better resin
- *Horizontal and Vertical Prototype Extrusion production in December and April*
  - *Integration Prototype Near Detector*
  - *Far Detector Full Scale Prototypes*

Stack of 28 (9-foot) Extrusions for IPND Segment Mechanical Prototype

September 19, 2006

Schedule

• Extrude N-24 and N-25	Oct 23 ‘06
– Verify rutile and anatase materials extrude with our die	
• Tune die with N-24	Oct 25- Nov 16 ‘06
– Depends on Greiner (die maker) technician’s availability	It takes about 2 weeks
• Order 70,000 lbs N-24 and 35,000 lbs N-25	Oct 25 ‘06
• Produce Horizontal Extrusions: IPND and Far Detector Prototypes	December ‘06
– Rutile (70,000 lbs)	
– Anatase (35,000 lbs)	
• Ship 16-cell die to Greiner to modify for Vertical Extrusions	Jan ’06
• Tune Die with N-25	Feb-Mar ’07
• Order N-25 and N-24 for Vertical Production	
• Produce Vertical Extrusions: IPND and Far Detector Prototypes	April ‘07